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⑤4発明の名称　エレクトロルミネツセンス素子及びその製造法

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明　細　書

1．発明の名称

エレクトロルミネッセンス素子及びその製造法

2．特許請求の範囲

（1）透明電極を形成した透明基体上に形成した厚さ0．1μmから2．0μmの少なくとも一種の蛍光体からなる薄膜発光層に、厚さ5μmから0．5mmの少なくとも一種の強誘電体粉末を適当な有機バインダー中に分散させてなる絶縁層が接し、その絶縁層側に対向電極を有する構造を特徴とする交流駆動エレクトロルミネッセンス素子。

（2）前記薄膜発光層が硫化物系蛍光体を含む少なくとも一種の蛍光体からなる単層膜もしくは、2種類以上の蛍光体による多層膜もしくは、蛍光体自体の発光効率を低下させない範囲で蛍光体以外の薄膜との多層膜である特許請求の範囲第1項記載の交流駆動エレクトロルミネッセンス素子。

（3）前記絶縁層が強誘電体粉末と常誘電体粉末の混合物もしくは、強誘電体粉末と抵抗体粉末の混合物である特許請求の範囲第1項記載の交流駆動エレクトロルミネッセンス素子。

（4）前記薄膜発光層と絶縁層の間に薄膜発光層内への電荷注入層として働く薄膜抵抗層を挿入してなる特許請求の範囲第1項記載の交流駆動エレクトロルミネッセンス素子。

（5）透明電極を形成した透明基体上に前記薄膜発光層を形成し、その上に、前記絶縁層を塗布もしくは成型し、さらにその上に、対向電極を連続して形成することを特徴とする交流駆動エレクトロルミネッセンス素子の製造法。

（6）透明電極を形成した透明基体上に形成した前記薄膜発光層と、導電体から成る対向電極上に塗布もしくは成型した前記絶縁層を別々に作成した後、薄膜発光層と絶縁層が接するように張り合わせて作製する特許請求の範囲第5項記載の交流駆動エレクトロルミネッセンス素子の製造法。

3．発明の詳細な説明

（産業上の利用分野）

本発明はエレクトロルミネッセンス素子及びそ

の製造法に関する。

（従来の技術）

一般にエレクトロルミネッセンス素子（以下、ＥＬ素子と記す）は、平面形固体発光表示素子として各種ディスプレイなどに利用されている。このＥＬ素子は構造上、薄膜形と分散形に分けられ、いずれも、その駆動方式から交流電圧駆動形と直流電圧駆動形に分けられるが、現在、薄膜形の交流二重絶縁構造ＥＬ素子及び分散形の交流駆動誘電体分散形ＥＬ素子が使用されている。

（発明が解決しようとする課題）

しかしながら、これらのＥＬ素子は高価で、駆動電圧が高く、十分な輝度を得るためには、通常、容易に得難い高周波電源を必要とするため、単相商用電源や直流電源を用いる表示ランプ等への応用が困難であった。さらに、薄膜形における高電圧印加によるカタストロフィックな絶縁破壊や分散形における短寿命の問題があった。このため、従来のＥＬ素子にあっては、薄膜形は高価なコンピュータ用端末ディスプレイ等限られた用途のみに使用され、分散形は十分な輝度と寿命が得られないため、その用途が極めて限られているのが現状である。

従って、本発明は、安価で、容易に製造でき、低電圧で、交流駆動でき、カタストロフィックな絶縁破壊の心配もなく、しかも大面積に高輝度な発光を示す長寿命なＥＬ素子を提供することを目的とする。

（問題点を解決するための手段）

本発明は、前期問題点を解決するための手段として、透明電極をつけた基体上に薄膜発光層、絶縁層および対向電極を付けた構造からなるエレクトロルミネッセンス素子において、前記薄膜発光層を硫化物系蛍光体を含む少なくとも一種の蛍光体からなる薄膜と、前記絶縁層を厚さ５μｍから０．５ｍｍの少なくとも一種の強誘電体からなる層で構成するようにしたものである。

本発明に係るＥＬ素子を構成する薄膜発光層は、硫化物系蛍光体を含む少なくとも一種の蛍光体材料を用い真空蒸着法、スパッタ法もしくは有機金属化学気相成長法で透明電極上に形成することができる。さらに、本発明に係るＥＬ素子を構成する強誘電体からなる絶縁層は、強誘電体粉末に適当な有機バインダーと粘弾性液体を混合したものを所定の厚さに加工し、乾燥して薄膜発光層上に形成できる。

本発明に係るＥＬ素子は、第１図に示すように基体１／透明電極２／薄膜発光層３／絶縁層４／対向電極５のような構造を持つが、透明電極をガラスあるいは有機フィルム等の透明な基体上に真空蒸着法、スパッタ法、スプレー法あるいは化学気相成長法等の公知の任意の方法によって形成し、その上に薄膜発光層を付けたものと、前記の絶縁層上に対向電極として真空蒸着法やスパッタ法で金属薄膜を作成するか、もしくは金属箔を張り付け、蛍光体発光層と絶縁層が接するように張り合わせることによりＥＬ素子を作製することが出来る。または、基体上の透明電極の上に薄膜発光層を形成した後、塗布法またはスクリーン印刷法等により前記絶縁層を形成し対向電極を付けてＥＬ素子を作製することも出来る。

また、薄膜発光層に効率的に電子を注入するため、第２図に示すように、薄膜発光層３と絶縁層４の間に電荷注入層６を形成するか、もしくは絶縁層４に導電性を付加しても良い。

（作用）

本発明に係るＥＬ素子は、薄膜発光層を作成する技術として高品質薄膜の形成が可能である分子線蒸着法、有機金属化学気相成長法ならびに原子層エピタキシー法等が使用できるため、結晶性が高品質な発光層を有する薄膜形ＥＬ素子の特徴である高い発光輝度および発光効率を実現でき、さらに絶縁層には、厚さ５μｍから０．５ｍｍの絶縁性が高く、一般に分散形ＥＬ素子で使用している高い誘電率をもつ強誘電体を使用しているため、薄膜形ＥＬ素子で問題となるカタストロフィックな絶縁破壊の心配もない。さらに、薄い発光層と誘電率の高い絶縁層を有するため、低電圧駆動が可能である。すなわち、薄膜形と分散形の長所を兼ね備えたＥＬ素子である。

以下、本研究の実施例について説明する。

（実施例１）

市販の錫ドープ酸化インジウム（ＩＴＯ）透明導電ガラス（ＨＯＹＡガラス製）上に、亜鉛ソースとしてジエチル亜鉛（ＤＥＺ）、イオウソースとして二硫化イオウ（CS_2）を用いた有機金属化学気相成長（ＭＯＣＶＤ）法により形成した硫化亜鉛（ＺｎＳ）薄膜に約６００℃で熱拡散法によりマンガン（Ｍｎ）をドープし、さらにその上に、チタン酸バリウム（$BaTiO_3$）粉末に無色透明な有機系のバインダーを主成分とする粘稠性液体を混ぜ室温で２時間混練して作成したペーストをスクリーン印刷法により約２０μｍの厚さに塗布し、１２０℃で約３０分間乾燥した後、アルミニウム（Ａｌ）金属薄膜を真空蒸着法により蒸着して対向電極としＥＬ素子を作製した。このＥＬ素子を５ｋＨｚの正弦波交流電圧で駆動していくと、約４０Ｖからオレンジ色の発光が認められ、最大輝度約１０００ｃｄ／ｍ²を得た。この場合のＥＬ素子の発光部の面積は約４００ｍｍ²であり、この発光は素子全面にわたって均一であった。得られた印加電圧に対する輝度特性を第３図に示す。

（実施例２）

実施例１と同様の方法でＩＴＯ透明導電ガラス上にＭｎをドープしたＺｎＳ：Ｍｎ薄膜発光層を形成したものと、対向電極としての厚さ約１００μｍのＡｌ板上に実施例１と同様の材料を混合して作成したペースト状の$BaTiO_3$をスクリーン印刷法により厚さ約２０μｍに塗布したものを、薄膜発光層と絶縁層が相接するようにして、線圧約１．８ｋｇｆ／ｃｍで１４０℃の温度で熱圧着してＥＬ素子を作製した。このＥＬ素子を５ｋＨｚの正弦波交流電圧で駆動していくと、約４０Ｖからオレンジ色の発光が認められ、最大輝度約１０００ｃｄ／ｍ²を得た。また発光は素子全面にわたって均一であった。

（実施例３）

実施例１と同様の方法でＩＴＯ透明導電ガラス上にＭｎをドープしたＺｎＳ：Ｍｎ薄膜発光層を形成しさらにその上に電荷注入層として厚さ約３０ｎｍの化学量論的組成比のずれた酸化タンタル（Ta_2O_5）薄膜抵抗層をスパッタ法により形成し、実施例１または２と同様の方法で絶縁層の$BaTiO_3$およびＡｌの対向電極を使用したＥＬ素子を作製した。このＥＬ素子を５ｋＨｚの正弦波交流電圧で駆動していくと、約３０Ｖから強いオレンジ色の発光が認められ、最大輝度約１５００ｃｄ／ｍ²を得た。また、発光は素子全面にわたって均一であった。

（実施例４）

実施例１および２において、ＩＴＯ透明導電ガラスの代わりに、コーニング７０５９ガラス上に高周波マグネトロンスパッタ法で作製したアルミニウムドープ酸化亜鉛（ＺｎＯ：Ａｌ）透明電極を用いて実施例１および２と同様にしてＥＬ素子を作製した。このＥＬ素子を５ｋＨｚの正弦波交流電圧で駆動したところ最高輝度約１５００ｃｄ／ｍ²を得た。また、発光は素子全面にわたって均一であった。

（実施例５）

実施例１～４において、ＺｎＳ：Ｍｎ薄膜発光層の代わりに、フッ化テリビウムドープ硫化亜鉛（ＺｎＳ：ＴｂＦ₃）薄膜発光層を用いて実施例１～４と同様にしてＥＬ素子を作製したところ、いずれの場合も素子全面にわたって均一な緑色の発光を得た。

（実施例６）

実施例１～４において、あらかじめ、透明電極を任意の文字にパターン化したＥＬ素子を作製した。このＥＬ素子を５ｋＨｚの正弦波交流電圧で駆動したところ、文字状にパターン化した透明電極下全面にわたって均一なオレンジ色発光を得た。

（実施例７）

実施例１～４において、透明電極と対向電極を互いに直角に対向するように、それぞれストライプ状にパターンニングした構造のＥＬ素子を作製した。このＥＬ素子を５ｋＨｚの正弦波交流電圧で駆動したところ、ドットマトリックス状にオレンジ色発光を得た。

（発明の効果）

本発明によれば、ＥＬ素子は絶縁層として厚さ５μｍから０．５ｍｍの強誘電体を使用しているため、従来の薄膜形ＥＬ素子で問題となるカタストロフィックな絶縁破壊による素子の破損の問題を解消できる。さらに、絶縁層が厚いため絶縁層を形成する際、望む粒径の強誘電体粉末を使用できる為、薄膜にくらべ高い誘電率をもつ絶縁層を得ることができ、素子の発光しきい値電圧の低減化が可能となっている。さらに発光層は、高品質な硫化物系蛍光体薄膜が作製可能なＭＯＣＶＤ法．原子層エピタキシ法、分子線蒸着などの真空蒸着法あるいはスパッタ法などを使って作成が可能なため、従来の分散形ＥＬ素子に比べ、高い発光輝度および発光効率が実現できる。また、透明電極を加工するだけで文字パターンの表示ができ、さらに、透明電極と対向電極が直角に対向するようにそれぞれストライプ状にパターンニングすることにより簡単にマトリックス表示が可能となる。すなわち、本発明のＥＬ素子は、従来の薄膜形ＥＬ素子と分散形ＥＬ素子の長所を兼ね備え、かつ、それぞれの短所を補ったこれまでに無いＥＬ素子といえる。

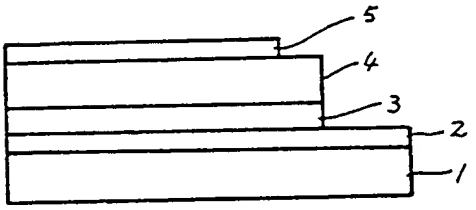
４．図面の簡単な説明

第１図は本発明に係るエレクトロルミネッセンス素子の構造の一例を示す断面図、第２図は本発明に係るエレクトロルミネッセンス素子の構造の他の実施例を示す断面図である。第３図は本発明に係るエレクトロルミネッセンス素子の電圧－輝度特性の一例である。

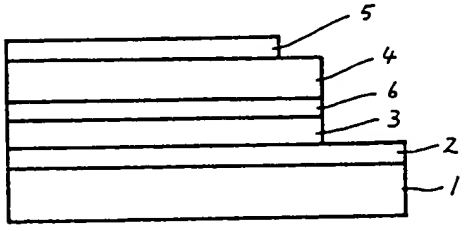
１～基体、２～透明電極、、３～薄膜発光層、４～絶縁層、５～対向電極、６～電荷注入層。

特許出願人　南　内嗣　外2名

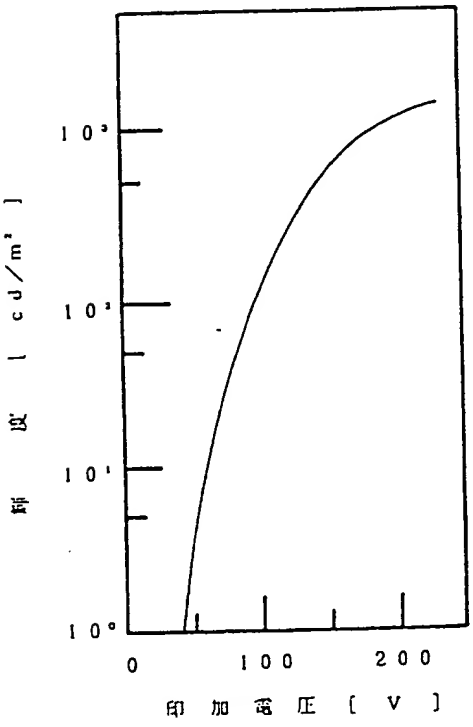
第　１　図



第　２　図



第　３　図



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Details

1. Name of invention

Electro luminescent element and the manufacturing method of the electro luminescent element

2. Range of the patent claims

(1) It is an AC drive electro luminescent element, which shall be characterized by having the structure, which is including the opposite electrode at the insulation layer side, which is made of at least one kind of ferroelectric powder, which shall have the thickness of between 5 μm and 0.5 mm and is scattered within the suitable organic binder, which shall be attached to the thin film emission layer, which is made of at least one kind of the phosphor material of the thickness between 0.1μm and 2.0 μm, which is created on the transparent base structure, and which shall form the transparent electrode.

(2) It is an AC drive electro luminescent element, which is mentioned in (1) of the range of the patent claim, and the abovementioned thin film emission layer shall be the single layer film, which is made of at least one kind of the phosphor material, which shall include sulfide related phosphor, or shall be the multi-layer, which is made of at least 2 kinds of the phosphor, or shall be the multi-layer, which is the multi-layer with the thin film, which is not the phosphor within the range of without lowering emission efficiency of the phosphor itself.

(3) It is an AC drive electro luminescent element, which is mentioned in (1) of the range of the patent claim, and the abovementioned insulation layer is either a mixture of ferroelectric powder and normal dielectric powder, or a mixture of ferroelectric powder and resistor powder.

(4) It is an AC drive electro luminescent element, which is mentioned in (1) of the range of the patent claim, which shall be characterized by inserting the thin film resistance layer, which shall work as the electric charge injection layer to the internal area of the thin film emission layer and placed between the abovementioned thin film emission layer and the insulation layer.

(5) It is a manufacturing method of the AC drive electro luminescent element, which shall be characterized by forming the abovementioned thin film emission layer on the transparent base structure, which shall form the transparent electrode, and the abovementioned insulation layer shall be molded or applied on top of the said thin film emission layer, and the opposite electrodes shall be formed consecutively on top of the said insulation layer.

(6) It is a manufacturing method of the AC drive electro luminescent element, which is mentioned in (5) of the range of the patent claim, and it shall be created by gluing in a way so that the thin film emission layer and the insulation layer shall be contacted after separately creating the abovementioned thin film emission layer, which is formed on the transparent base structure, which shall form the transparent electrode, and the abovementioned insulation layer, which is either molded or applied onto the opposite electrodes, which shall be composed of the conductive part.

3. Detailed explanation of the invention
(Utility field of the industry)
This invention is concerning the electro luminescent element and the manufacturing method of the electro luminescent element.

(Existing technique)
Commonly, the electro luminescent element (hereinafter referred to as EL element) is used for various kinds of displays as the flat solid emission display element. Such El element can be divided into the thin film type and the scattered type for the purpose of the structure, and they can be divided into the AC voltage driving type and DC voltage driving type for the purpose of the driving method. However, currently the thin film style AC double insulation structure EL element and the scattered style AC drive dielectric scattered EL element are used.

(The problem to be solved by the invention)
However, these EL elements are expensive and because the driving voltage is high and so that in order to obtain sufficient luminance, generally it requires a high frequency power source, which is not easy to obtain, and therefore, it is difficult to apply for the use of the display lamp, etc., which shall use a single-phase power source or DC power source, etc. Further, there are problems such as catastrophic insulation destruction from applying high voltage concerning the thin film type, and the life of the scattered type is rather short. Therefore, concerning the existing EL element, the thin film type is used within the limited area, such as an expensive terminal display for computers, and the scattered type is used also within an extremely limited area due to the reasons that the life is short and it is not able to obtain the sufficient luminance.

Therefore, the purpose of this invention is to provide the EL element, which shall have a long life, emission with high luminance in larger areas without having concerns of catastrophic insulation destruction, as well as providing low cost, easy to manufacture, low voltage EL element, which uses the AC drive.

(The method of how to solve the problem)
In order to solve the aforementioned problems, in this invention the electro luminescent element is structured by the thin film emission layer on the base structure, which shall have the transparent electrodes and the insulation layer with the opposite electrodes. The abovementioned thin film emission layer shall be composed of the thin film, which shall have at least one kind of the phosphor material including sulfide related phosphor, and the abovementioned insulation layer shall be made of at least one kind of a film, which is composed of ferroelectric, which shall have the thickness of between 5μm and 0.5 mm.

The thin film emission layer, which shall construct the EL element concerning the invention, can be created on the transparent electrode using at least one kind of phosphor material including the sulfide related phosphor either by the vacuum deposition technique, sputtering method or the organic metallic chemical vapor deposition method. Further, the insulation layer, which is made of ferroelectric that shall construct the EL

element, which is related to this invention, can be created on the thin film emission layer by drying after processing the mixture of mixing the suitable organic binder and viscosity liquid with the ferroelectric powder to the designated thickness.

The EL element which is related to this invention shall have the structure, which is shown in Figure 1 such as the base structure 1 / transparent electrode 2 / thin film emission layer 3 / insulation layer 4 / opposite electrode 5. However, the EL element can be created by forming using one of the well known methods of the vacuum deposition technique, the sputtering method, the spray method or the chemical vapor deposition method, etc., and then either by creating the metallic thin film by the vacuum deposition method or the sputtering method as the opposite electrode on the abovementioned insulation layer together with the thin film emission layer, or by firmly attaching (gluing) the metallic foil in order to make the phosphor emission layer and the insulation layer contacted to each other by gluing together. It is also possible to create the EL element by attaching the opposite electrode to the abovementioned insulation layer, which is formed either by the applying method or the screen printing method, etc. after forming the thin film emission layer on the transparent electrode, which is on the base structure.

Also, in order to insert the electron in the thin film emission layer efficiently, as shown in Figure 2, the electric charge injection layer 6 can be created between the thin film emission layer 3 and the insulation layer 4, or conductivity can be added to the insulation layer 4.

(Function)
The El element which is related to this invention is able to use the molecular beam deposition method, the atomic layer epitaxy method and the organic metallic chemical vapor deposition method, etc., which can create the high quality thin films for the purpose of the technique to create the thin film emission layer, and therefore, high emitting luminance and high emitting efficiency, which is the strong point of the thin film type EL element, which shall have the emission layer with the high quality crystallization, can be actualized. Further, because the ferroelectric with high dielectric constant, which is commonly used for the scattered type EL element for the purpose of the insulation layer with high insulation ability, which shall have the thickness of between 5 μm and 0.5 mm, such problem among the thin film type EL element, which is the catastrophic insulation destruction, shall not occur. Further, because it shall contain the thin emission layer and the insulation layer with high dielectric constant, low voltage drive can be possible. Therefore, this EL element shall have both advantages of the thin film type and the scattered type.

Hereinbelow, the implementation example of this research shall be explained.

(Example of implementation 1)
Manganese (Mn) was doped by the thermal diffusion method at approximately 600 °C to the zinc sulfide (ZnS) thin film, which was created by the organic metallic chemical vapor deposition (MOCVD) method, which used CS_2 as the sulfur source and DEZ as the zinc source, on the ITO transparent conductive glass (HOYA manufactured glass), which

is a good on the market. And on top of this, the paste, which is created by the mixture of the viscosity liquid, which shall have the transparent and colorless organic binder, and $BaTiO_3$ powder, which was mixed at room temperature for 2 hours, was applied by the screen printing method for the approximate thickness of 20 μm, then it was dried for 30 min. at 120 °C, and after that the EL element was created by making the opposite electrode from the aluminum (Al) metallic thin film using the vacuum deposition technique. By driving this EL element at the sine wave AC voltage of 5kHz, orange color emitting was recognized from around 40V, and obtained approximately 1000 cd / m^2 as the highest luminance. The emitting area of the EL element of this case was approximately 400 mm^2, and this emission was even on the entire surface of the element. The luminance characteristic for the obtained applied voltage is shown in Figure 3.

(Example of implementation 2)
The EL element was created by the thermocompression bonding at 140°C and the wire pressure of approximately 1.8 kgf / cm making the thin film emission layer and the insulation layer to be attached, which was applied between the ZnS : Mn thin film emission layer, which Mn was doped, and which was created on ITO transparent conductive glass by the same method of the implementation example 1, and $BaTiO_3$, which is the paste, which was applied for the approximate thickness of 20 μm by the screen printing method, which was created by the mixture of the same material as the implementation example 1 on the Al board of the approximate thickness of 100 μm as the opposite electrode. By driving this EL element at the sine wave AC voltage of 5kHz, orange color emitting was recognized from around 40V, and obtained approximately 1000 cd / m^2 as the highest luminance. Also, this emission was even on the entire surface of the element.

(Example of implementation 3)
Using the same method of the implementation example 1, ZnS : Mn thin film emission layer, which Mn was doped, was created on the ITO transparent conductive glass, and on top of this, Ta_2O_5 thin film resistance layer with shifted stoichiometric composition rate of the thickness of approximately 30 nm was created by the sputtering method for the purpose of the electric charge injection layer, and then following the same method of the implementation example 1 or the implementation example 2, the insulation layer $BaTiO_3$ and the EL element, which shall use the opposite electrode of Al, were created. By driving this EL element at the sine wave AC voltage of 5kHz, strong orange color emitting was recognized from around 30V, and obtained approximately 1500 cd / m^2 as the highest luminance. Also, this emission was even on the entire surface of the element.

(Example of implementation 4)
Concerning the implementation example 1 and the implementation example 2, the EL element was created by the same method as the implementation example 1 and the implementation example 2 using ZnO : Al transparent electrode, which was created by the high frequency magnetron sputtering method on the corning 7059 glass, which was used instead of the ITO transparent conductive glass. By driving this EL element at the sine wave AC voltage of 5kHz, the highest luminance of approximately 1500 cd / m^2 was obtained. Also, this emission was even on the entire surface of the element.

(Example of implementation 5)
Concerning the implementation examples 1 to 4, the EL element was created by the same method as the implementation examples 1 to 4 using ZnS : TbF_3 thin film emission layer instead of ZnS : Mn thin film emission layer, and in all cases, the green color emitting was obtained on the entire surface of the element evenly.

(Example of implementation 6)
Concerning the implementation examples 1 to 4, the EL element, which shall have the transparent electrode, which is patterned into a voluntary character in advance, was created. By driving this EL element at the sine wave AC voltage of 5kHz, the orange color emitting was obtained evenly on the entire surface of the transparent electrode, which was patterned into a character style.

(Example of implementation 7)
Concerning the implementation example of 1 to 4, the EL element was created, which shall have the structure of the striped patterning in order to make the transparent electrode and the opposite electrode to be facing each other at right angles. By driving this EL element at the sine wave AC voltage of 5kHz, orange color emitting was obtained in a dot matrix style.

(Effectiveness of the invention)
Concerning this invention, due to the reason that the EL element shall use the ferroelectric of the thickness of between 5 μm and 0.5 mm for the purpose of the insulation layer, the problem with the existing thin film style EL element, which is a damage of the element occurred by the catastrophic insulation destruction, shall be dissolved. Further, because the insulation layer is thick, and therefore, when creating the insulation layer, ferroelectric powder of the desired particle size can be used, which shall make it possible to obtain the insulation layer, which shall have higher dielectric constant compared to the thin film, and reducing of the emitting threshold voltage of the element shall become possible. Further, because the emission layer can be created by the vacuum deposition technique such as the MOCVD method, the atomic layer epitaxy method and the molecular beam deposition method, etc., or by the sputtering method, which are able to create high quality sulfide related phosphor thin film, therefore, compared to the existing scattered type EL element, higher emitting luminance and higher emitting efficiency can be actualized. Also, just by processing the transparent electrode, the character pattern can be displayed, and further, by the striped patterning in order to make the transparent electrode and the opposite electrode to be facing each other at right angles, matrix display can easily be actualized. Therefore, this EL element of the invention shall have both advantages of the thin film type and the scattered type, and also, it makes up for the shortcomings of the both types, which can be said as the unprecedented EL element.

4. Simple explanation of figures
Figure 1 shows a cross section figure of an example structure of the electro luminescent element, which is related to this intention, and figure 2 shows a cross section figure of

another implementation example of the structure of the electro luminescent element, which is related to this invention. Figure 3 is an example of voltage – luminance characteristics of the electro luminescent, which is related to this invention.

1. Base structure
2. Transparent electrode
3. Thin film emission layer
4. Insulation layer
5. Opposite electrode
6. Electric charge injection layer

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Figure 1 (P.518, left upper)

Figure 2 (P.518, left bottom)

Figure 3
Vertical: Luminance (cd / m^2)
Horizontal: Applied voltage (V)